IS 10243 (1993): 2, 4-D Ethyl Ester EC [FAD 1: Pesticides and Pesticides Residue Analysis]

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IS 10243 : 1993
(Reaffirmed 2012)

भारतीय मानक

2, 4-डी इथाईल एस्टर – विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

2, 4-D ETHYL ESTER EC — SPECIFICATION

(First Revision)

UDC 632·954 : 661·725-11/-12

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

January 1993

Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Division Council.

2, 4-D ethyl ester EC is used as a weedicide.

The material is generally manufactured to contain 38 percent (*m/m*) 2,4-D ethyl ester having 34 percent (*m/m*) of 2,4-D acid.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*).' The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2, 4-D ETHYL ESTER EC — SPECIFICATION

(Frist Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2,4-D ethyl ester EC.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
1448 [P : 20] : 1984	Methods of test for petroleum and its products : [P : 20] Flash point by Abel apparatus (*first revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
7233 : 1991	2,4-D ethyl ester, technical (*second revision*)
8190 (Part 2) : 1988	Requirement for packing of pesticides: Part 2 Liquid pesticides (*second revision*)
10627 : 1983	Methods for sampling of pesticidal formulations

3 REQUIREMENTS

3.1 Constituents

3.1.1 The material shall consist of 2,4-D ethyl ester, technical, together with suitable solvent(s), emulsifier(s) and other formulant(s).

3.1.2 2,4-D ethyl ester, technical employed in the formulation of this material shall conform to IS 7233 : 1991 .

3.2 Description

The material shall be clear liquid, free from sediment. Suspended matter shall be negligible. It shall readily form an emulsion on dilution with water, suitable for spray.

3.3 Identity Test

When determined by the method prescribed in Annex G of IS 7233 : 1991 the retention time of the 2,4-D ethyl ester present in the sample shall be the same as that of reference standard 2,4-D.

3.4 Physical

The material shall comply with physical requirements specified in **3.4.1** to **3.4.4.**

3.4.1 *Cold Test*

No turbidity or separation of solid and/or oily matter or both shall occur when the material is subjected to cold test at 10°C as prescribed in **13.1** of IS 6940 : 1982 or any other lower temperature as agreed to between the purchaser and the supplier.

3.4.2 *Flash Point (Abel)*

When determined by the method prescribed in IS 1448 (P : 20) : 1984, the flash point of the material shall be above 24·5°C.

3.4.3 *Emulsion Stability*

Any separation including creaming at the top and sedimentation at the bottom of 100 ml of emulsion prepared in standard hard water with 2·0 ml concentrate, shall not exceed 2·0 ml when tested by the method prescribed in **13.3** of IS 6940 : 1982.

3.4.4 *Heat Stability*

After treating in accordance with the method prescribed in **13.4** of IS 6940 : 1982, the material shall comply with the requirements specified in **3.2**, **3.4.3** and **3.5.**

NOTE — The material need not be put to heat treatment, if the product has crossed half of its shelf life, as ascertained from the date of manufacture and date of expiry declared on the container.

3.5 Chemical

The material shall comply with the chemical requirements specified in **3.5.1** and **3.5.2.**

3.5.1 *2,4-D Ethyl Ester Content*

When determined by the method prescribed in Annex A, the observed 2,4-D ethyl ester content percent (*m/m*) of any of the samples shall not differ from the declared nominal

value by more than the percent tolerance limits indicated below:

Nominal Value, Percent	*Tolerance Limits, Percent*	
Up to 9	+10 − 5	of the nominal value
Above 9 and below 50	± 5	
50 and above	+ 5 − 3	

3.5.2 *Free* 2,4-D

When tested by the method prescribed in Annex B of IS 7233 : 1991 free acidity (as 2,4-D acid) shall not be more than 4 percent by mass.

4 PACKING

The containers shall be packed according to the requirements given in IS 8190 (Part 2) : 1988.

5 MARKING

The containers shall bear legibly and indelibly the following information in addition to any other information required under the *Insecticides Act*, 1968 and Rules:

a) Name of the material;

b) Name of the manufacturer;

c) Batch number;

d) Date of manufacture and date of expiry;

e) Net mass of contents;

f) Nominal 2,4-D ethyl ester content, percent (*m/m*); and

g) A cautionary notice worded as in the *Insecticides Act*, 1968 and Rules.

6 SAMPLING

When material in bulk quantity is offered for inspection, representative samples shall be drawn and tested for conformity as prescribed in IS 10627 : 1983 within 90 days of its manufacturing. When the material is offered for inspection after 90 days of its manufacturing, samples shall be drawn as prescribed in IS 10627 : 1983. However, the criteria for conformity shall be the limits of tolerances as applicable over the declared nominal value and given under **3.5.1** of this standard.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in **3.3** to **3.5.2**.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 3.5.1)

DETERMINATION OF 2, 4-D ETHYL ESTER CONTENT

A-1 GENERAL

Total extractable 2,4-D content, percent by mass shall be estimated by the method specified under **A-3**. Free 2,4-D content, percent by mass shall be estimated by the method specified in Annex B of IS 7233 : 1991. 2,4-D ethyl ester content shall be then calculated by multiplying the difference between the total extracted 2,4-D and free 2,4-D by the factor 249/221.

A-2 REAGENTS

A-2.1 Potassium Hydroxide — Pellets.

A-2.2 Isopropanol

A-2.3 Petroleum Ether

A-2.4 Phenolphthalein Indicator — 1 percent solution in 96 percent ethanol (*m/m*).

A-2.5 Hydrochloric Acid — 1 : 1 (*v/v*).

A-2.6 Ammonium Hydroxide — 1 : 1 (*v/v*).

A-2.7 Barium Chloride Solution — 0·1 percent aqueous solution (*m/v*).

A-2.8 Methyl Orange Indicator — 0·1 percent aqueous solution (*m/v*).

A-2.9 Diethyl Ether

A-2.10 Silver Nitrate Solution — 5 percent (*m/m*).

A-2.11 Ethanol — Neutral, alternatively methanol, neutral may be used.

A-2.12 Sodium Hydroxide Solution — 0·1 N.

A-2.13 Bromothymol Blue Indicator Solution

0·04 percent solution in alcohol (*m/m*).

A-3 PROCEDURE

A-3.1 Weigh accurately an amount of the sample equivalent to about 1 g of 2,4-D into a round-bottom flask, add about 1·5 g of potassium hydroxide pellets, 80 ml isopropanol, 20 ml distilled water and 2-3 glass beads. Fit a reflux condenser and heat vigorously under reflux for about an hour.

A-3.2 Cool, filter the contents of the flask, wash the flask thoroughly with 10 ml portions of distilled water 6 to 7 times or until traces of alkali are removed when tested by phenolphthalein indicator solution. Filter and collect the filtrate into a 500-ml separatory funnel. Extract the contents of the separatory funnel with two 50 ml portions of petroleum ether to remove unsaphonifiable oils. Carefully draw off the aqueous phase into a 500-ml beaker and evaporate it on a water bath, slowly and carefully, until the volume is reduced to about 50 ml.

A-3.3 Cool and transfer the solution into a 250-ml volumetric flask, wash the beaker 2 to 3 times with small portions of distilled water and transfer the washings also to the volumetric flask. Add a few drops of hydrochloric acid until the disappearance of alkalinity. Add ammonium hydroxide and make the solution alkaline and check with phenolphthalein indicator. Carefully add 3 ml of barium chloride and shake continuously to precipitate any fatty acid derived from the saponification. Dilute the solution to the mark with distilled water and allow to stand for about 5 minutes with occasional stirring.

A-3.4 Filter the contents of the volumetric flask into a 250-ml beaker. Take out with pipette 100 ml of filtrate into a 250-ml separatory funnel and using methyl orange indicator acidify with hydrochloric acid. Extract the 2,4-D acid with three 25 ml portions of diethyl ether. After each extraction with ether, allow sufficient time for the layers to separate. Combine the three ether extracts and wash with successive portions of distilled water till free of chlorides (about 10-15 ml washings should not give any precipitate with silver nitrate). Transfer the contents of the separatory funnel into a 500-ml conical flask, wash the separatory funnel with 2 small portions of ether, collect the washings also into the conical flask. Add 80 ml ethanol, and titrate with standard sodium hydroxide solution using bromothymol blue indicator.

A-3.5 Carry out a blank determination with 75 ml ether and 80 ml ethanol.

A-4 CALCULATION

A-4.1 Total extractable 2, 4-D content, percent by mass $= \dfrac{(V_1 - V_2) \times N \times 2{\cdot}5 \times 22{\cdot}1}{M}$

where

V_1 = volume, in ml, of standard sodium hydroxide solution required for the test;

V_2 = volume, in ml, of standard sodium hydroxide solution required for the blank determination;

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of sample taken for the test.

A-4.2 2, 4-D ethyl ester content $= (A - B) \times \dfrac{249}{221}$

where

A = Total extractable 2,4-D content (*see* **A-4.1**); and

B = Free 2,4-D content (*see* **3.5.2**).

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Bureau of Indian Standards

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(First Revision)

(*Page* 1, *clause* **3.4.4**) — Delete.

(FAD 1)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 JANUARY 1998
TO
IS 10243 : 1993 2, 4-D ETHYL ESTER EC — SPECIFICATION

(First Revision)

(*Page* 3, *clause* A-4.2) — Substitute the following for the existing matter:

'A-4.2 2, 4-D ethyl

$$\text{ester content} = A \times \frac{249}{221}$$

where

A = total extractable 2, 4-D content.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India